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<b>(51) International Patent Classification <sup>6</sup> :</b> <b>C22C 19/05</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 99/67435</b> <b>(43) International Publication Date:</b> 29 December 1999 (29.12.99)
<b>(21) International Application Number:</b> PCT/EP99/04285 <b>(22) International Filing Date:</b> 21 June 1999 (21.06.99)  <b>(30) Priority Data:</b> 09/103,097                      23 June 1998 (23.06.98)                      US  <b>(71) Applicants:</b> SIEMENS AKTIENGESELLSCHAFT [DE/DE]; Wittelsbacherplatz 2, D-80333 München (DE). HOWMET RESEARCH CORPORATION [US/US]; 405 Steamboat Road, Greenwich, CT 06830 (US).  <b>(72) Inventors:</b> ESSER, Winfried; Am geraden Weg 39, D-44805 Bochum (DE). PAUL, Uwe; Oranienstrasse 9, D-52066 Aachen (DE). MAYR, Christoph; Drosselweg 3, D-83064 Raubling (DE). CORRIGAN, John; 1710 Calthrop Neck Road, Yorktown, VA 23693 (US). VOGT, Russel, G.; 302 Blue Heron Drive, Yorktown, VA 23692 (US). MIHAL- ISIN, John, R.; 545 Mountain Avenue, Caldwell, NJ 07006 (US). BENNETT, John, K.; 5 Critchards Woodbury, Exeter EX5 1PE (GB).  <b>(74) Common Representative:</b> SIEMENS AKTIENGE- SELLSCHAFT; Postfach 22 16 34, D-80506 München (DE).		<b>(81) Designated States:</b> IN, JP, RU, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> DIRECTIONALLY SOLIDIFIED CASTING WITH IMPROVED TRANSVERSE STRESS RUPTURE STRENGTH		
<b>(57) Abstract</b>  Directionally solidified columnar grain nickel base alloy casting consisting essentially of, in weight %, of about 11.6 % to 12.70 % Cr, about 8.50 to 9.5 % Co, about 1.65 % to 2.15 % Mo, about 3.5 % to 4.10 % W, about 4.80 % to 5.20 % Ta, about 3.40 to 3.80 % Al, about 3.9 % to 4.25 % Ti, about 0.05 % to 0.11 % C, about 0.003 % to 0.015 % B, balance essentially Ni and having substantial transverse stress rupture strength and ductility as compared to a similar casting without boron present.		

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DIRECTIONALLY SOLIDIFIED CASTING WITH  
IMPROVED TRANSVERSE STRESS RUPTURE STRENGTH

FIELD OF THE INVENTION

The present invention relates to nickel base superalloy castings and, more particularly, to directionally solidified (DS) nickel base superalloy castings having a columnar grain microstructure and substantially improved transverse stress rupture strength and ductility.

BACKGROUND OF THE INVENTION

U.S. Patent 4 597 809 describes single crystal castings made from a nickel base superalloy having a matrix with a composition consisting essentially of, in weight %, of 9.5% to 14% Cr, 7% to 11% Co, 1% to 2.5% Mo, 3% to 6% W, 1% to 4% Ta, 3% to 4% Al, 3% to 5% Ti, 6.5% to 8% Al + Ti, 0 to 1% Nb, and balance essentially nickel with the matrix containing about 0.4 to about 1.5 volume % of a phase based on tantalum carbide as a result of the inclusion in the alloy of about 0.05% to about 0.15% C and extra Ta in an amount equal to 1 to 17 times the C content.

Single crystal castings produced from the aforementioned nickel base superalloy exhibit inadequate transverse grain boundary strength. The present inventors attempted to produce directionally solidified (DS) columnar grain castings of the nickel base superalloy. However, the directionally solidified (DS) columnar grain castings produced were unacceptable as DS castings as a result of the castings exhibiting essentially no transverse grain boundary

strength and no ductility when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi). The transverse grain boundary strength and ductility were so deficient as to render DS columnar grain castings produced from the aforementioned nickel base superalloy unsuitable for use as turbine blades of gas turbine engines.

An object of the present invention is to provide DS columnar grain castings based on the aforementioned single crystal nickel base superalloy having substantially improved transverse stress rupture strength and ductility to an extent that the DS castings are acceptable for use as turbine blades of a gas turbine engine.

Another object of the present invention is to provide such DS columnar grain castings based on the aforementioned single crystal nickel base superalloy having substantially improved transverse stress rupture strength and ductility without adversely affecting other mechanical properties and corrosion resistance of the DS castings.

#### SUMMARY OF THE INVENTION

The present invention involves including boron in the nickel base superalloy described hereabove in a manner discovered to significantly improve transverse stress rupture strength and ductility of directionally solidified (DS) columnar grain castings produced from the boron modified superalloy. In accordance with the present invention, boron is added to the aforementioned superalloy composition in an effective amount to substantially improve

transverse stress rupture strength and ductility of directionally solidified columnar grain castings produced from the boron-modified superalloy. The boron concentration preferably is controlled in the range of about 0.003% to about 0.015% by weight of the superalloy composition to this end. In conjunction with addition of boron to the superalloy composition, the carbon concentration preferably is controlled in the range of about 0.05% to about 0.11% by weight of the superalloy composition.

A preferred nickel base superalloy in accordance with an embodiment of the present invention consists essentially of, in weight %, of about 11.6% to 12.70% Cr, about 8.50 to 9.5% Co, about 1.65% to 2.15% Mo, about 3.5% to 4.10% W, about 4.80% to 5.20% Ta, about 3.40 to 3.80% Al, about 3.9% to 4.25% Ti, about 0.05% to 0.11% C, about 0.003% to 0.015% B, and balance essentially Ni. The boron modified nickel base superalloy can be cast as DS columnar grain castings pursuant to conventional DS casting techniques such as the well known Bridgeman mold withdrawal technique.

DS castings produced in this manner typically have a plurality of columnar grains extending in the direction of the principal stress axis of the casting with the <001> crystal axis generally parallel to the principal stress axis. DS columnar grain castings pursuant to the present invention preferably exhibit a stress rupture life of at least about 150 hours and elongation of at least about 2.5% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) and will find use as

turbine blades, vanes, outer air seals and other components of a industrial and aero gas turbine engines.

The above objects and advantages of the present invention will become more readily apparent from the following detailed description taken with the following drawings.

#### DESCRIPTION OF THE DRAWINGS

Figure 1A is a photomicrograph at 11.25X taken transverse to the longitudinal axis of a DS cast specimen showing the columnar grain microstructure.

Figures 1B, 1C and 1D are similar photomicrographs at 50X, 100X and 200X, respectively, of the columnar grain microstructure of Figure 1A.

#### DETAILED DESCRIPTION OF THE INVENTION

The present invention involves including boron in a particular nickel base superalloy in a manner discovered to unexpectedly and surprisingly provide significantly enhanced transverse stress rupture strength and ductility of DS columnar grain castings produced from the boron-modified superalloy. The nickel base superalloy which is modified pursuant to the present invention is described in U.S. Patent 4 597 809, the teachings of which are incorporated herein by reference. A nickel base superalloy in accordance with an embodiment of the invention consists essentially of, in weight %, of about 9.5% to 14% Cr, about 7% to 11% Co, about 1% to 2.5% Mo, about 3% to 6% W, about 1% to 6% Ta, about 3% to 4% Al, about 3% to 5% Ti, about 0 to 1% Nb, and balance essentially Ni

and B present in an amount effective to substantially improve transverse stress rupture strength of a DS casting as compared to a similar casting without boron present.

The present invention modifies the aforementioned nickel base superalloy to include boron in the alloy in an amount discovered effective to provide substantial transverse stress rupture strength and ductility of a DS columnar grain casting produced from the alloy as compared to a similar casting without boron present. Preferably, the nickel base superalloy is modified by the inclusion of boron in the range of about 0.003% to about 0.015%, preferably 0.010% to 0.015%, by weight of the superalloy composition to this end. In conjunction with addition of boron to the superalloy composition, the carbon concentration is controlled in a preferred range of about 0.05% to about 0.11% by weight of the superalloy composition. The transverse stress rupture strength and ductility of DS castings produced from the boron modified nickel base superalloy are provided to an extent that the castings are rendered acceptable for use as turbine blades and other components of gas turbine engines.

A particularly preferred boron-modified nickel base superalloy casting composition in accordance with the present invention consists essentially of, in weight %, of about 11.6% to 12.70% Cr, about 8.50 to 9.5% Co, about 1.65% to 2.15% Mo, about 3.5% to 4.10% W, about 4.80% to 5.20% Ta, about 3.40 to 3.80% Al, about 3.9% to 4.25% Ti, about 0.05% to 0.11% C, about 0.003% to 0.015% B, and

balance essentially Ni and castable to provide a DS columnar grain microstructure. The DS microstructure of the columnar grain casting, Figure 1A, typically includes about 0.4 to about 1.5 volume % of a phase based on tantalum carbide shown as light gray particles in Figures 1B, 1C and 1D. Some of the light gray particles in the DS microstructure appear to be eutectic gamma prime phase. The somewhat rounded dark features dispersed throughout the DS microstructure in Figures 1A through 1D comprise voids present in the particular cast specimens examined. Although not wishing to be bound by any theory, it is thought that boron and carbon tend to migrate to the grain boundaries in the DS microstructure to add strength and ductility to the grain boundaries at high service temperatures, for example, 816 degrees C (1500 degrees F) typical of gas turbine engine blades.

DS columnar grain castings produced from the above boron-modified nickel base superalloy in accordance with the present invention typically have the <001> crystal axis parallel to the principal stress axis of the casting and exhibit a stress rupture life of at least about 150 hours and elongation of at least about 2.5% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) applied perpendicular to the <001> crystal axis of the casting.

For example, the following DS casting tests were conducted and are offered to further illustrate, but not limit, the present invention. A heat #1 having a nickel base superalloy composition in



accordance with the aforementioned U.S. Patent 4 597 809 and heats #1A and #2 of boron modified nickel base superalloy in accordance with the present invention were prepared with the following compositions, in weight percentages, set forth in Table I:

TABLE I

Heat	Cr	Co	Mo	W	Ta	Al	Ti	C	B	Ni
#1	12.1	9.0	1.8	3.7	5.2	3.6	4.0	0.7	0.001	balance
#1A	12.1	9.0	1.8	3.7	5.2	3.6	4.0	0.8	0.010	balance
#2	12.1	9.0	1.8	3.7	5.2	3.6	4.0	0.9	0.011	balance

Each heat was cast to form DS columnar grain non-cored castings having a rectangular shape for transverse stress rupture testing pursuant to ASTM E-139 testing procedure. The DS castings were produced using the conventional Bridgeman mold withdrawal directional solidification technique. For example, each heat was melted in a crucible of a conventional casting furnace under a vacuum of 1 micron and superheated to 1427 degrees C (2600 degrees F). The superheated melt was poured into an investment casting mold having a facecoat comprising zircon backed by additional slurry/stucco layers comprising zircon/alumina. The mold was preheated to 1482 degrees C (2700 degrees F) and mounted on a chill plate to effect unidirectional heat removal from the molten alloy in the mold. The melt-filled mold on the chill plate was withdrawn from the furnace into a solidification chamber of the casting

furnace at a vacuum of 1 micron at a withdrawal rate of 6-16 inches per hour. The DS columnar grain castings were cooled to room temperature under vacuum in the chamber, removed from the mold in conventional manner using a mechanical knock-out procedure, heat treated at 1250 degrees C (2282 degrees F) for 4 hours, analyzed for chemistry, and machined to specimen configuration. Stress rupture testing was conducted in air at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) applied perpendicular to the <001> crystal axis of the specimens.

The results of stress rupture testing are set forth in TABLE II below where LIFE in hours (HRS) indicates the time to fracture of the specimen, ELONGATION is the specimen elongation to fracture, and RED OF AREA is the reduction of area of the specimens to fracture. The BASELINE data corresponds to test data for Heat #1, and the #1A and #2 data corresponds to test data for Heat #1A and #2, respectively. The BASELINE data represent an average of two stress rupture test specimens, while the #1A and #2 data represent a single stress rupture test specimen.

TABLE II

ALLOY	# OF TESTS	TEMPERATURE C (F)	STRESS Mpa (KSI)	LIFE (HRS)	ELONGATION (%)	RED OF AREA (%)
BASELINE	2	750 (1382)	660 (95.7)	0	0	0
#2	1	750 (1382)	660 (95.7)	182	2.6	6.3
#1A	1	750 (1382)	660 (95.7)	275	3.1	4.7

It is apparent from TABLE II that the DS columnar grain specimens produced from heat #1 exhibited in effect essentially no (e.g. zero

hours stress rupture life) transverse grain boundary strength when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi). That is, the specimens failed immediately to provide an essentially zero stress rupture life. Moreover, the elongation and reduction of area data were essentially zero. These stress rupture properties are so deficient as to render the DS columnar grain castings produced from heat #1 unacceptable for use as turbine blades of gas turbine engines.

In contrast, TABLE II reveals that DS columnar grain specimens produced from heat #1A exhibited a stress rupture life of 275 hours, an elongation of 3.1%, and a reduction of area of 4.7 % and specimens from heat #2 exhibited a stress rupture life of 182 hours, an elongation of 2.6%, and a reduction of area of 6.3% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi). These stress rupture properties of the invention represent an unexpected and surprising improvement over those of specimens produced from heat #1 and render DS columnar grain castings produced from heats #1A and #2 more suitable for use as turbine blades and other components of gas turbine engines.

The present invention is effective to provide DS columnar grain castings with substantial transverse stress rupture strength and ductility. These properties are achieved without adversely affecting other mechanical properties, such as tensile strength, creep strength, fatigue strength, and corrosion resistance of the

DS castings. The present invention is especially useful to provide large DS columnar grain industrial gas turbine (IGT) blade castings which have the alloy composition described above to impart substantial transverse stress rupture strength and ductility to the castings and which have a length of about 20 centimeters to about 60 centimeters and above, such as about 90 centimeters length, used throughout the stages of the turbine of stationary industrial gas turbine engines. The above described boron-modified nickel base superalloy casting composition can be cast as DS columnar grain or single crystal components.

While the invention has been described in terms of specific embodiments thereof, it is not intended to be limited thereto but rather only to the extent set forth in the following claims.

## CLAIMS:

## WE CLAIM

1. A directionally solidified columnar grain nickel base alloy casting, consisting essentially of, in weight %, of about 9.5% to 14% Cr, about 7% to 11% Co, about 1% to 2.5% Mo, about 3% to 6% W, about 1% to 6% Ta, about 3% to 4% Al, about 3% to 5% Ti, about 0 to 1% Nb, and balance essentially Ni and B present in an amount effective to substantially improve transverse stress rupture strength of said casting as compared to a similar casting without boron present.
2. The casting of claim 1 wherein B is present in the range of about 0.003% to about 0.015% by weight.
3. The casting of claim 1 that has a stress rupture life of at least about 150 hours and elongation of at least about 2.5% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) applied in a direction perpendicular to a <001> crystal axis of said casting.
4. The casting of claim 1 which is gas turbine engine blade having a length of about 20 centimeters to about 90 centimeters.

5. A directionally solidified columnar grain nickel base alloy casting consisting essentially of, in weight %, of about 11.6% to 12.70% Cr, about 8.50 to 9.5% Co, about 1.65% to 2.15% Mo, about 3.5% to 4.10% W, about 4.80% to 5.20% Ta, about 3.40 to 3.80% Al, about 3.9% to 4.25% Ti, about 0.05% to 0.11% C, about 0.003% to 0.015% B, balance essentially Ni and having substantially improved transverse stress rupture strength as compared to a similar casting without boron present.

6. The casting of claim 5 that has a stress rupture life of at least about 150 hours and elongation of at least about 2.5% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) applied perpendicular to a <001> crystal axis of said casting.

7. A directionally solidified columnar grain nickel base alloy casting having a nominal composition consisting essentially of, in weight %, of about 12.00% Cr, about 9.00% Co, about 1.85% Mo, about 3.70% W, about 5.10% Ta, about 3.60% Al, about 4.00% Ti, about 0.0125% B, about 0.09% C, balance essentially Ni and having a stress rupture life of at least about 150 hours and elongation of at least about 2.5% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) applied perpendicular to a <001> crystal axis of said casting.

8. A method of making a directionally solidified casting, comprising casting an alloy consisting essentially of, in weight %, of about 9.5% to 14% Cr, about 7% to 11% Co, about 1% to 2.5% Mo, about 3% to 6% W, about 1% to 6% Ta, about 3% to 4% Al, about 3% to 5% Ti, about 0 to 1% Nb and balance essentially Ni and B in an amount effective to substantially improve transverse stress rupture strength into a mold, and directionally solidifying the alloy in the mold to form a columnar grain casting having substantially improved transverse stress rupture strength by virtue of the inclusion of boron in said alloy as compared to a similar casting without boron present.

9. The method of claim 8 wherein B is included in an amount of about 0.003% to about 0.015% by weight.

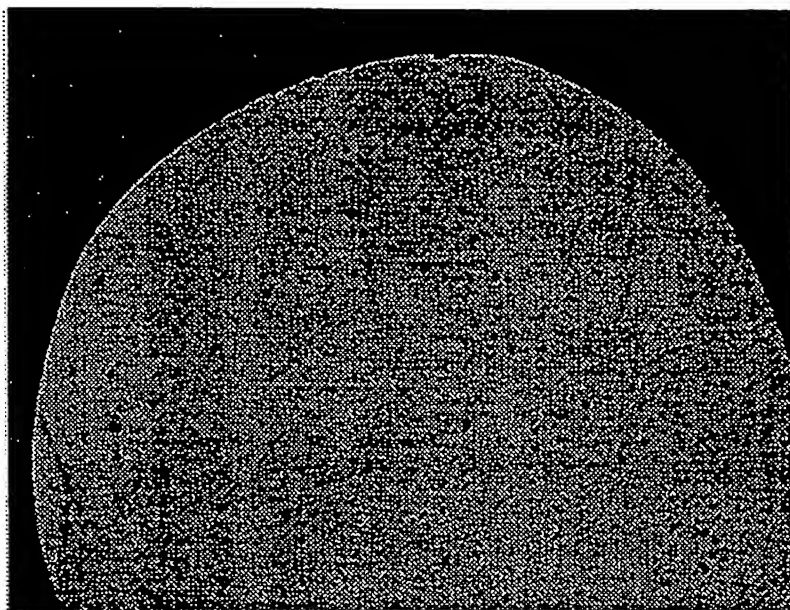
10. The method of claim 8 wherein the directionally solidified casting has a stress rupture life of at least about 150 hours and elongation of at least about 2.5% when tested at a temperature of 750 degrees C (1283 degrees F) and stress of 660 Mpa (95.7 Ksi) applied perpendicular to a <001> crystal axis of said casting.

11. A method making a directionally solidified casting, comprising providing a nickel base alloy consisting essentially of, in weight %, of about 11.6% to 12.70% Cr, about 8.50 to 9.5% Co, about 1.65% to 2.15% Mo, about 3.5% to 4.10% W, about 4.80% to 5.20% Ta, about

3.40 to 3.80% Al, about 3.9% to 4.25% Ti, about 0.05% to 0.11% C, about 0.003% to 0.015% B, and balance essentially Ni, casting the alloy into a mold, and solidifying the alloy in the mold to form a casting having a directionally solidified columnar grain microstructure having substantially improved transverse stress rupture strength as compared to a similar casting without boron present.

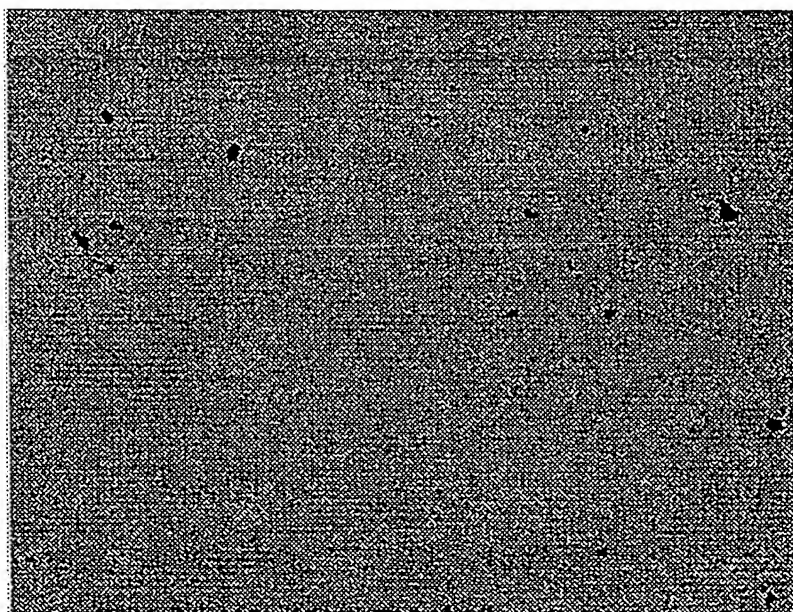
12. Nickel base alloy consisting essentially of, in weight %, of about 11.6% to 12.70% Cr, about 8.50 to 9.5% Co, about 1.65% to 2.15% Mo, about 3.5% to 4.10% W, about 4.80% to 5.20% Ta, about 3.40 to 3.80% Al, about 3.9% to 4.25% Ti, about 0.05% to 0.11% C, about 0.003% to 0.015% B, balance essentially Ni.





X 11.25

FIG 1A



X 50

FIG 1B

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FIG 1C

X 100

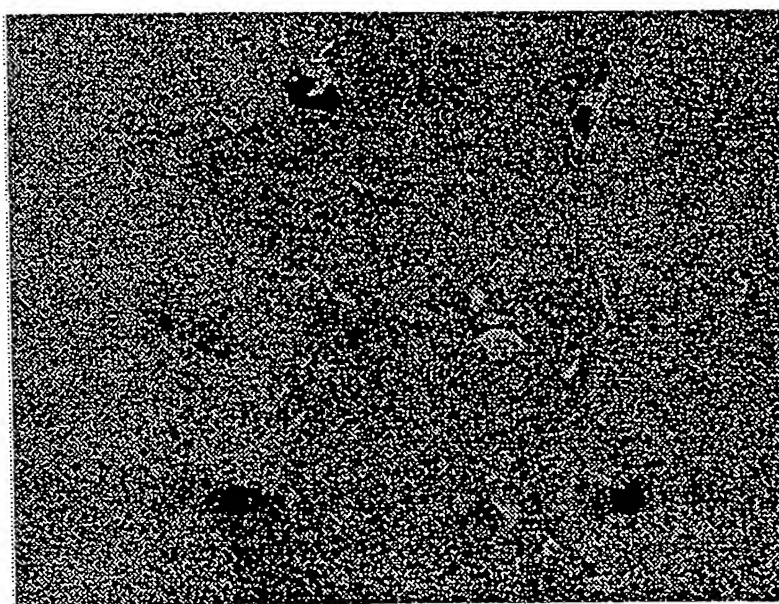


FIG 1D

X 200

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## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 99/04285

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C22C19/05

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## B. FIELDS SEARCHED

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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